INVESTIGATIVE MONITORING - COLUMBIA RIVER

This report presents data accumulated during FY-78 to meet commitments to Annual work Plan assignments (FY 78-79) to the Marrowstone Field Station Fisheries Assistance Office, Nordland, Washington.

The work was conducted under the Environmental Contaminant Evaluation Program, Activity 1110 Work Element 220, Project 03.

ABSTRACT

White sturgeon (Acipenser transmontanus) were sampled in four river sections of the Columbia River; below Bonneville Dam, in Lake Bonneville, Lake Umatilla, and Lake Wallula, to determine if PCB and pesticide residues are present in concentrations high enough to affect reproductive capability, the marketability or home consumptive use. A total of 22 tissue samples and 2 roe samples were subjected to residue analyses for PCB's and organochlorines. The results revealed presence of PCB's, DDT, DDE, DDD, Dieldrin, HCB, &BHC, T-Nonachlor, &-Chlordane, and Y-Chlordane in some or all of the tissues anal-.yzed. PCB and DDE were found in all tissues and in the roe. PCB and DDE in tissues and percent lipids in Lake Wallula sttransfer were significantly (.05) greater than those taken below Bonneville Dam and in Lake Bonneville. The concentrations of PCB and DDE appear to be positively correlated with percentage of lipids. Mean concentrations of PCB's and organochlorine compounds appear to be below the FDA action level of $5~\mathrm{ug/g}$ with respect to consumptive use. PCB levels in sturgeon roe are highly suspect with respect to possible influence on the reproductive potential of sturgeon when compared to published data relative to other species.

A response monitoring program was initiated to determine the levels of PCB and pesticides in sturgeon (Acipenser transmontanus) in the mid and lower Columbia River. This monitoring was predicated by National Pesticide Monitoring data which indicated that some fish species in the mid-Columbia River region contained excessive amounts of PCB, DDT and its metabolites. This excess was noted in an administrative report entitled "Organochlorines and Heavy Metals Detected in Fish- A Partial Review of the Fish and Wildlife Service Contrib-

ution to the National Pesticide Monitoring Program- 1967-1973", by David F. Walsh, October 1975. Sampling stations specifically mentioned in this report were: #44 Yakima River, Granger, Washington; #97 Columbia River, Pasco, Washington; and #46 at Bonneville, Oregon.

As a result of the review, a memorandum was issued on January 14, 1976, by the Program Coordinator, Environmental Contaminant Evaluation, which recommended "that each Regional Pesticide Specialist develop, as needed, investigative monitoring projects (either fish and/or birds) for his region within the limitations stated."

The fish species analyzed in the mid-Columbia River for the National Pesticide Monitoring Program data were, for most part, bottom feeding suckers and carp. The sturgeon is also primarily a bottom feeder and as such would be subject to similar PCB and pesticide exposure.

Also, because of its size, up to 1,000 pounds, and longevity of 30-plus years it would appear to represent the end of a food chain and an excellent bio-accumulator of water-borne contaminants.

The sturgeon is classified as a food fish by the Washington Department of Fisheries. In past years it was considered incidental to the salmon harvest. However, reductions in the commercial salmon fishing seasons in the Columbia River have encouraged the development of a commercial set—line fishery. In 1976 the commercial harvest in the lower river section (Bonneville to Astoria) was 723,000 pounds. This poundage was amassed in some 22,500 fish in the four to six-foot lengths which is the commercial size restriction. During this same

year some 24,000 pounds were taken from the mid-Columbia (Ronneville Dam to McNary Dam).

A sturgeon sport fishery also occurs in the Columbia River. The lower river fishermen catch is from 10,000 to 15,000 fish annually. These fish are in the three to six-foot lengths which is the sport fish size restriction. The mid-Columbia sport catch is unknown at this time. However, in view of the findings and trecommendations of the National Pesticide Monitoring Program it was deemed desireable to determine if PCB and pesticide residues in sturgeon are present in concentrations high enough to affect their reproductive capabilaty, marketability or home consumptive use by sportsmen.

SAMPLE COLLECTION PROCEDURES

Specimens were collected in some areas by use of set lines which were authorized under a Scientific Collecting Permit issued by the State of Washington Department of Fisheries. Other samples were collected by accompanying commercial fishermen into the field during the set-line fishing season, and from a commercial fish processing plant at Astoria, Oregon. Tissues collected consisted of edible filets weighing not less than 100 grams. These tissues were taken from the dorsal-lateral area immediately posterior to the operculum. The filets were skinned, wrapped in aluminum foil and a tag affixed noting the date, location, length, and sex. The tissues were placed in a cooler containing dry ice and frozen as quickly as possible. All samples were stored in a freezer and maintained in a frozen condition until analyzed. A section of the first bony ray of the pectoral fin was collected for age determination, tagged in a manner

similar to the tissue sample, and frozen.

SAMPLE SITES

with the exception of the lower river section (Bonneville to Astoria) the Columbia River is essentially a series of pools formed by hydroelectric dams. These dams are believed to have eliminated all but occasionally passage of upstream migrating sturgeon. Consequently, except for possible downstream drift of juveniles, each pool contains an isolated population. These isolated populations necessitated sampling by individual pool and the lower river section to obtain information relative to the individual groups. Sampling by individual pool also increased the probability of detecting sources of PCB and pesticides entering in waters of tributary streams.

Below Bonneville (Bonneville Dam to Astoria)

This section of the Columbia (Fig. 1) is free flowing from Bonneville to the mouth, and is about 127 miles long. In addition to contaminants in the main stem Columbia coming from above Bonneville Dam and industries located along the banks within the sample section it has a number of tributaries which could contribute water-borne contaminants. These tributaries are; the Cowlitz and Lewis Rivers from the State of Washington, and the Sandy and Willamette Rivers from the State of Oregon. Specific sample sites in this section are unknown. All samples were collected from a commercial fish food processing plant located at Astoria, Oregon. A total of eight filet and one eng sample was taken from this section. Of the eight filets taken

Lake Bonneville (Bonneville Dam to the Dalles Dam)

TABLE 1 — Columbia River sturgeon filet and roe samples by river section, age, sex, and length data.

Sample No.	Age	FOX.	længt!	
•	**		Total (in)	Fork (in)
Below Bonneville - Jul	v 1977			
12 roe sample	<u> </u>	F	57.00	
73	12	UK	48.50	43.00
14	10	UK	47.50	42.50
15 (Duplicate of 14)				- 100 Mg 25F .
16	16	UK	52.25	47.00
17 (Duplicate of 16)			War the sale than topy	
18	13	UK	55,00	51.00
19 (Duplicate of 18)			-	- *
20	10	UK	47.00	40.50
Lake Bonneville - Febr				
	7	percent of the second of the s	28.75	26.00
2 (Duplicate of 1)	w. 9W-	3.1	.	
3	15	M 	51.00	47.75
4	6	T.M	28,25	25.00
5	9	Ŧ	37.00	32.25
6	7	IM	29.75	26.50
7	24	F	76 . 00	70.50
8 (Duplicate of 7)				
9	21	N	71.75	63.00
10 (Duplicate of 9)				
ll roe sample from fis	h #7			
Lake Umatilla - July 1	078			
50	20	₹*** \$4	70.00	64.50
51	9	,] } }	35.25	
52	13	, го М	32.23 49.00	31,00
53	9	г) М		43.00
54		r. F	37.25	33.25
,344	13	3'	46.25	42,00
Lake Wallula - June 19	78	-		
21	19	M	64.00	58,25
22	12	M	47.25	42.50
23	17	A. C.	60.00	52,25
24	13	M	52.75	47.00
25	15	М	56.00	50.50

M - Mala

F - Female

TM - Immature

CX - Unknown

This sample section (Fig. 2) is about 47 miles in length. The major tributaries entering this section are the Wind, Little White Salmon and Klickitat Rivers from the State of Washington. Hood River and Mill Creek enter from the State of Oregon, A total of ten filet samples were taken from this section, and three of this total number were duplicates (Table 1). One egg sample was collected in this section along with filet sample #7 and duplicate #8. The majority of the fish taken in this section were caught about 1 mile downstream from the Hood River Marina (Fig. 2). In addition to the above samples seven sturgeon and two carp were collected by the Vancouver Fisheries Assistance Office and submitted to a private laboratory in Seattle for analyses. These data are included in the discussion.

This sample section (Fig. 3) is about 75 miles in length. Tributary streams are Rock Creek from the State of Washington and the John Day River, Willow, Sixmile and Butter Creeks from the State of Oregon. A total of five filet samples were taken from this section (Table 1). All samples were collected from a side arm arm channel of the main stem Columbia approximately 10 miles west of the town of Umatilla, Oregon.

Lake Wallula (McNary Dam to Priest Rapids Dam)

This sample section (Fig.4) is about 100 miles in length. Major tributaries entering this section are the Walla Walla, Yakima, and Snake Rivers. The Walla Walla and Yakima Rivers flow through highly developed agricultural areas. A total of five filets were collected in this section (Tablel). The actual sampling site was in the

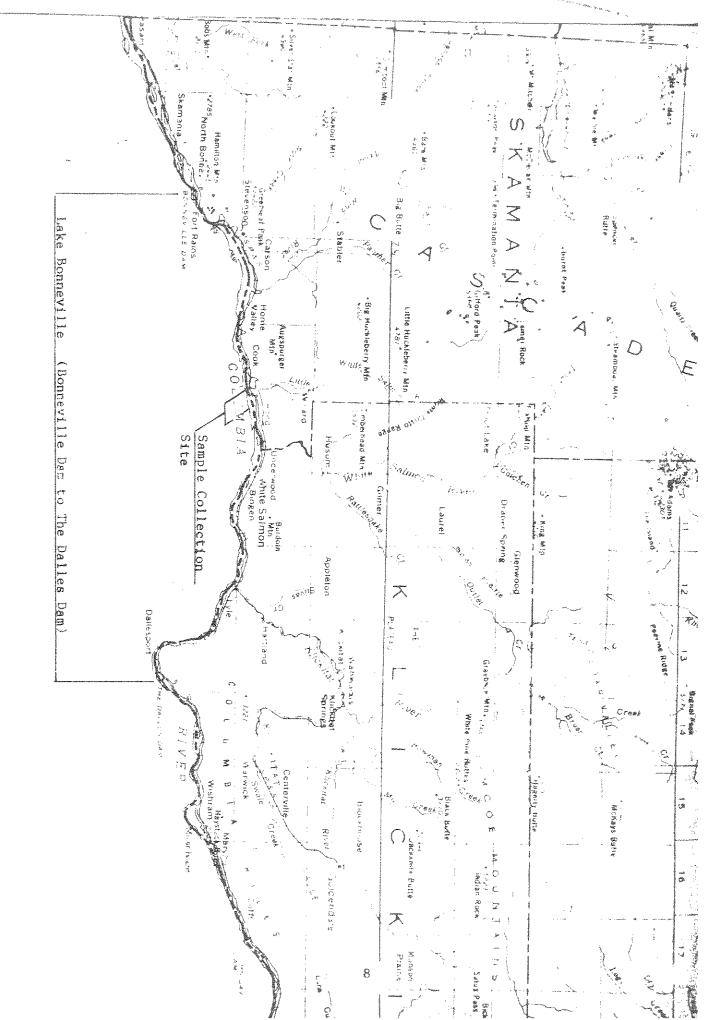
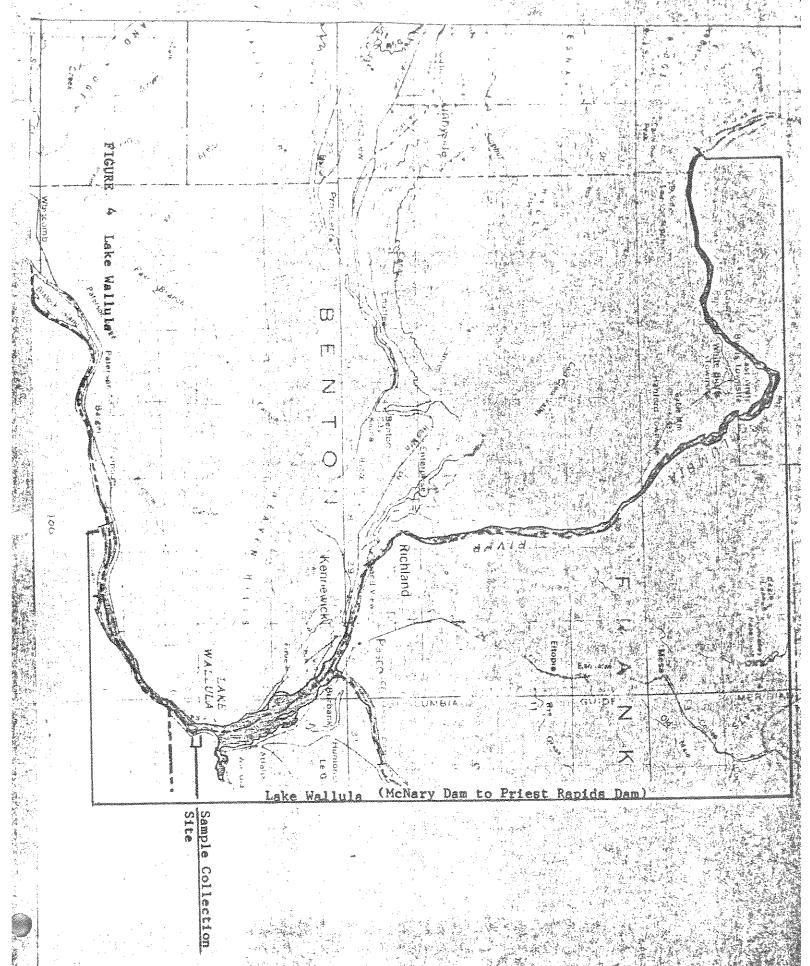


FIGURE 2 Lake Bonneville

FIGURE 3 Lake Umatilla



immediate vicinity of Port Kelly Station which is approximately

10 miles below the Tri-Cities Area of Richland, Kennewick and

Pasco. The three major tributaries also enter the Columbia River within this ten mile distance.

ANALYTICAL PROCEDURES

All sturgeon filets and roe samples were shipped, packed in dry ice via commercial airline, to a laboratory under contract to the Fish and Wildlife Service. The techniques utilized by this laboratory were as follows;

Methodology: Limit of detection: 0.05 ppm for Chlorinated Insecticides 0.10 ppm for PCB's and toxaphene.

Storage: All samples were stored in a deep freeze prior to analysis

Preparation: All flesh samples were ground in a Hobart food grinder, Model A-200. The egg sample was homogenized in a Waring Blender cup.

(Flesh and egg) 25 grams of sample was weighed into a Extraction: 150 ml beaker. 100 grams of anhydrous sodium sulfate was mixed into the sample. The mixture was transferred to a Waring blender cup. An additional 100 grams of sodium sulfate was added to the beaker. The sodium sulfate was transferred to the blender cup. 150 mls of petroleum ether was added to the blender cup and blended at medium speed for two minutes. The petroleum ether was decanted through a 100 mm powder funnel plugged with glass wool into a 500 ml erlenmeyer. 125 mls of petroleum ether was added to the blender cup and blended for two minutes and decanted through the glass wool powder funnel. An additional 125 mls of petroleum ether was added to the blender cup, blended, and decanted. The remaining mixture left in the blender cup was transferred to the powder funnel. The blender cup was rinsed with 50 mls of petroleum ether and decanted through the powder funnel. The resulting solution was concentrated to approximately 5 ml on a steam bath and made to 25 ml with petroleum other.

Clean-up:

(Flesh) A 5 ml aliquot of the extract was placed on previously standardized florisil and eluted with 150 ml of 3% ethyl ether in petroleum ether followed by 250 ml of 15% ethyl ether in petroleum ether. The resulting solutions were concentrated to approximately 5 mls and made to 25 ml with petroleum ether. The first elution was injected on a gas chromatograph to determine if silicic acid separation was required. The second elution was injected for quantitation of Dieldrin and Endrin. All samples required silicic acid separation.

(Egg) A 10 ml aliquot of the original extract was taken to dryness and made up to 25 ml with 25% Toluene in Ethyl Acetate. A 5 ml aliquot was put through pre-standardized G.P.C. The resulting solution was concentrated to 1-2 mls on a flash evaporator and made to 25 ml with petroleum ether. Λ 10 ml aliquot was taken for silicic acid separation.

Silicic Acid Separation: A 10 ml aliquot of the first elution from florisil was transferred to a pre-standardized Silicar C-4 column and eluted with 80 mls of petroleum ether, followed by 350 petroleum ether followed by 150 mls of 1% acetonitide, 19% llexane, 80% methylene chloride mixture. The resulting solutions were concentrated on a flash evaporator to approximately 2 ml and made to 10 mls with petroleum ether. 10 microliters or less of each elution were injected into a gas chromatograph for quantitation.

Lipid determination: A 5 ml aliquot of the original extract was transferred to a pre-weighed 2 dram vial. The solvent was removed and the vial was placed in a 40° C oven for 24 hours. The vial was removed, dessicated, re-weighed and the amount of lipid was calculated.

Gas Chromatography: Instrument: Hewlett Packard Model 5710A equipped with linerized N163, automatic injector and 3352C Data system.

Column (1) For all chlorinated pesticides and PCB's except chlordane isomers

Packing: 1.5/1.95% ov-17/QF-1 on 80/100 mesh Supelcoport.

Column: Glass 6' x 4 mm ID glass

Column Temperature: 200° C Injector Temperatura: 250° C Detector Temperature: 300° C

Carrier Gas: 95% Argon 5% Methane Flow: 33 ml/minute or adjusted to give DDE a retention time of

approximately 10.0 minutes.

Column (2) Quantitation of chlordane isomers

Packing: 3% OV-1 on 80/100 Supelcoport

Column: 6' 4 mm ID glass Column Temperature: 190° C Injector Temperature: 250° C Detector Temperature: 300° C

Carrier gas: 95% Argon 5% Methane

Flow: 32 ml/minute

AGE DETERMINATION

The pectoral bony rays collected during tissue sampling were used for age determination. These rays were immersed in a commercial decalcifying solution for 24 hours, washed and sectioned with a freezing microtome. The sections were stained in hematoxylin to bring out the growth rings and mounted on slides. A binocular microscope was used to read these sections.

RESULTS

The following organochlorine compounds were identified in some or all of the 22 sturgeon filets sampled: polychlorinated biphenyls (PCB's reported as Arochlor 1254), dichlorodiphenyltrichloroethane (DDT) and its metabolites DDE and DDD, dieldrin, hexachlorobenzene (HCB), benzene hexachloride (QBHC), and three chlordane related compounds, achlordane, ychlordane, and t-nonachlor. Mean concentrations of these compounds in sturgeon are shown in Figure 5.

The percentage of sturgeon that contained detectable quantities of each compound are shown in Figure 6. The three chlordane compounds were detected only in sturgeon from Lake Bonneville.

The compounds BCB and QBBC were identified only in Lake Wallula

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fish. Dieldrin was detected in one sturgeon from Lake Bonneville and four from Lake Wallula.

In the DDT group, mean DDE concentrations were more than twice that of DDD in all areas sampled and were 10-35 times that in the three lakes sampled above Bonneville Dam. Mean DDE concentrations increased steadily from a low of .08 ppm below Bonneville Dam to a high of 1.77 ppm in Lake Wallula sturgeon. Concentrations in Lake Wallula sturgeon were significantly (.05) higher than those below Bonneville Dam and in Lake Bonneville.

Mean concentrations of PCB's followed a pattern similar to that of DDE, increasing from a low of .21 ppm below Bonneville Dam to a high of 1.63 ppm in Lake Wallula. As with DDE, PCB concentrations in Lake Wallula sturgeon were significantly (.05) greater than those from below Bonneville Dam and in Lake Bonneville.

In addition to the sturgeon filets, two egg samples were analyzed.

One sample came from a 57 inch (TL) sturgeon taken below Bonneville

Dam in February 1978. DDE and PCB's were detected at 0.07 and 0.16

ppm respectively and the percentage of lipids was measured at 6.52.

The other sample came from a 76 inch (TL) fish taken in Lake Bonneville in July 1977. This sturgeon contained the following organochlorines in ppm: PCB's, 1.45; DDT, 0.47; DDE, 1.75; DDD, 0.71;

dieldrin, 0.14; t-nonachlor, 0.16; cchlordane, 0.78; and cchlordane,

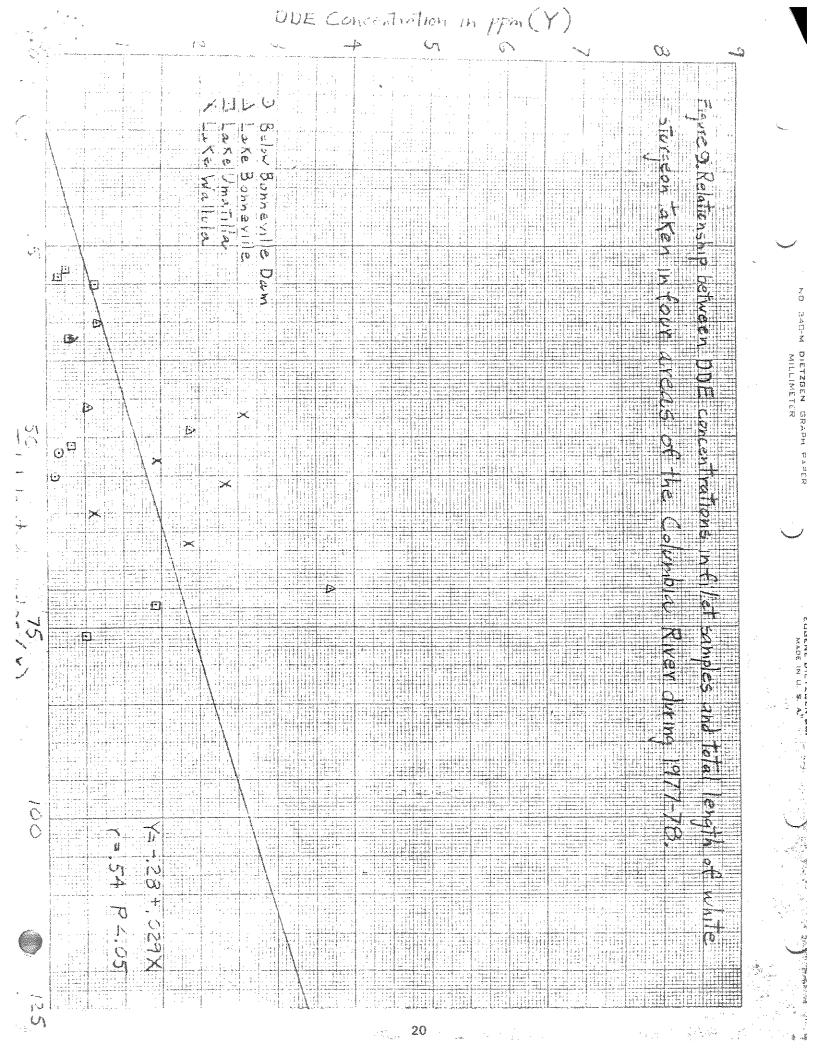
0.33. The egg sample contained 38.3 percent lipids.

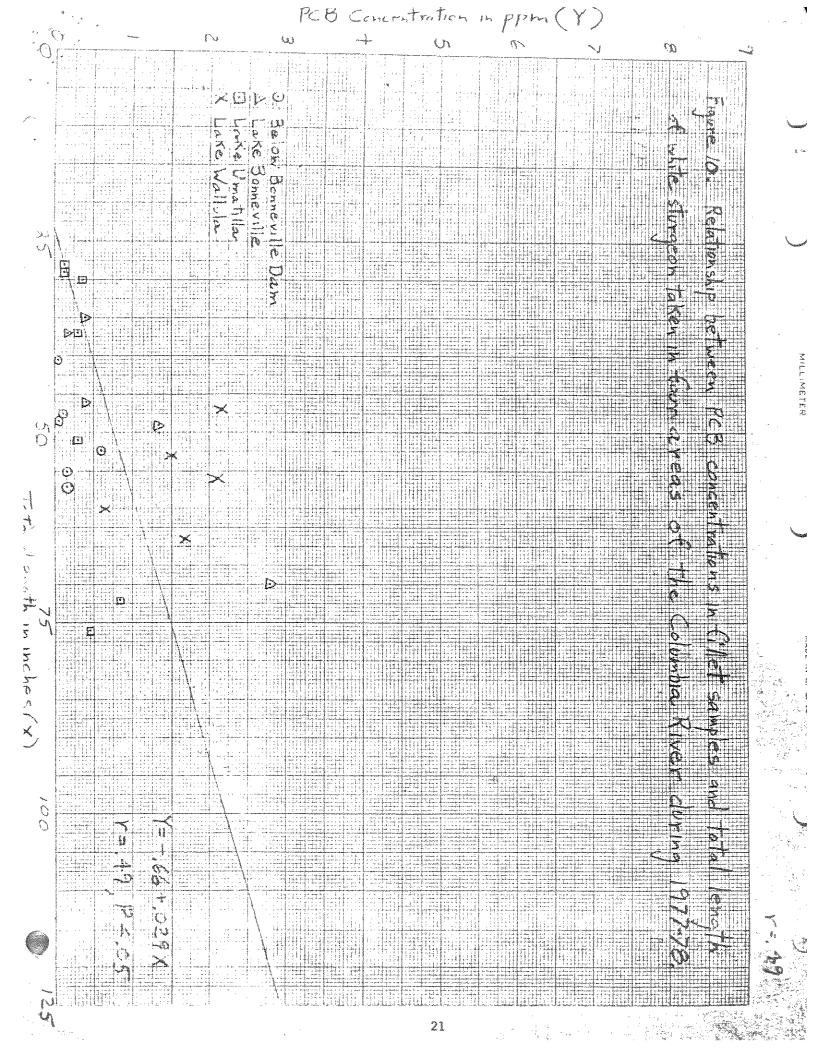
The mean percentage of lipids in sturgeon filets also increased

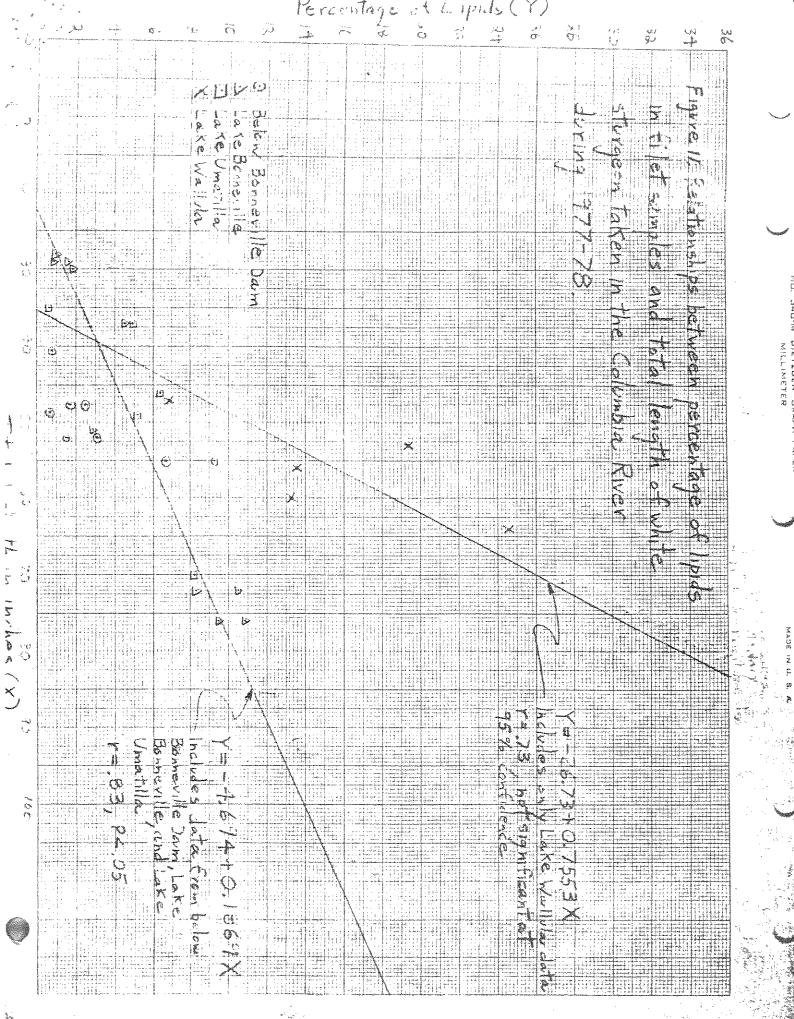
as samples were examined in an upstream manner. Lipids in sturgeon taken below Bonneville Dam averaged 2.8 percent, from Lake Bonneville 4.4 percent, from Lake Umatilla 5.1 percent, and from Lake Wallula 15.6 percent. The Lake Wallula average is significantly (.05) greater than those from below Bonneville Dam and Lake Bonneville.

The concentration of DDE was positively correlated (r = .45.P < .05)to the percentage of lipids in sturgeon filets (Figure 7). Similarly, PCB concentrations and lipid percentages were positively correlated (r =.53,P <.05), (Figure 8), DDE and PCB concentrations were also positively correlated with sturgeon length (r = .54.P < .05 and r = .49, P < .05 respectively) (Figures 9 and 10). The fact that both sturgeon length and percentage of lipids were positively correlated with DDE and PCB concentrations is explained by the positive correlation between length and percentage of lipids (Figure 11). Because data from Lake Wallula sturgeon did not coalesce with data from other areas two separate regressions were calculated. In both instances correlations were positive. Lake Wallula exhibited a sharp increase (slope = .76) in percentage of lipids when length increased. However, due to the small sample size, the correlation coefficient r z.73 was not significantly (.05) different from zero. Combined sturgeon data from other areas showed a positive but smaller increase in lipids per increase im length (slope = 19, r = 83, P < .05). Although the regression of DDE and PCB concentrations against percent

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lipids (Figures 7 and 8) shows a positive relationship, it is recognized that other variables such as sex, length, age, and area taken may influence the lipid - concentration relationship. Therefore, an attempt was made to eliminate all other variables from the relationship by examining five sturgeon in which duplicate samples were taken. Because each lipid value is associated with its own set of organochlorine concentrations, the ratio of the lipid values may be plotted against the ratio of organochlorines concentrations. Figure 12 shows that the ratio of compound concentrations was positively correlated (r= .67, P <.05) with the ratio of lipid percentages. The linear regression yields the equation $\frac{2}{x} = 134 + .67 \frac{x}{x_e}$ where X, is the larger of the two lipid percentages, Y; the corresponding compound concentration of that sample, and X_2 and Y_2 the lipid percentage and compound concentration of the duplicate sample. This equation can be used to adjust the organochlorine concentration associated with a certain lipid percentage to one associated with any value desired. The equation appears to be valid for egg samples as well as filets. In Table 2, duplicate samples from each of two sturgeon are compared before and after adjustment to a common lipid percentage. The value of 7.09 percent was chosen because it represents the mean lipid value for the 22 sturgeon collected, but any value could be chosen. As Table 2 shows, the concentration of DDE and PCB's are closer after adjustment than before. Also the concentrations of DDE and PCB's in the egg sample approximate those of the filets.

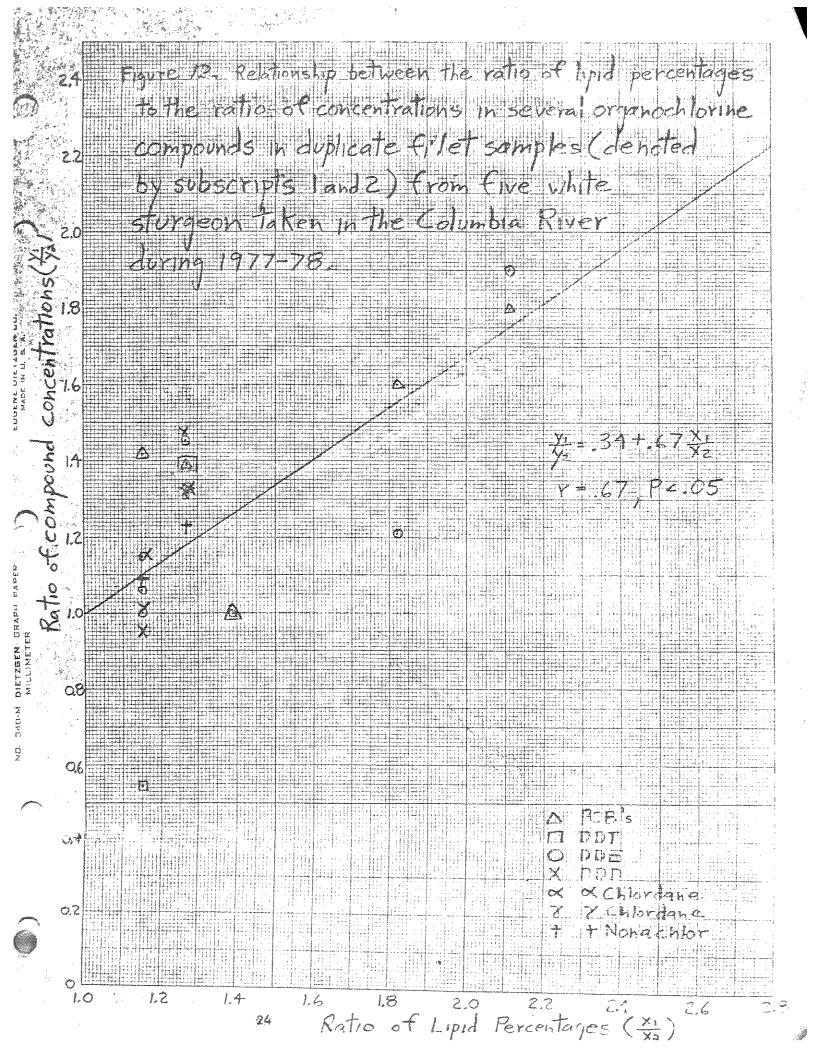


TABLE 2 — Comparison of DDE and PCB concentrations in duplicate samples before and after adjustment to a common lipid percentage. Adjustment was made using the equation $\frac{1}{12} = \frac{34}{34} + \frac{1}{16} \cdot \frac{7}{12} = \frac{34}{16} \cdot \frac{1}{16} \cdot \frac{1$

Sturgeon sampled	Unad	justed	data	A	djuste	d data
	Lipids	DDE	PCB	Lipids	DDE	PCB
	%	ppm	ppm	%	ppm	ppm
76 in. Lake Bonneville						
Filet sample #7	10.90	0,51	0.54	7.09	0.37	0.39
Filet sample #8	9.47	0.48	0.38	7.09	0.39	0.31
Egg sample #11	38.30	1.75	1.45	7.09	0.44	0.37
52.25 in. below Bonneville				-		
Dam						
Filet sample #16	3.14	0.21	0.78	7.09	0.39	1.45
Filet sample #17	1.49	0.11	0.43	7.09	0.39	1.52

Table 3 compares the mean concentrations of DDE and PCB's as they occur in unadjusted data (Figure 5) to data adjusted to the common lipid value of 7.09 percent. Because concentrations of DDE and PCB's were not detectable in some sturgeon taken below Bonneville Dam, a valid comparison cannot be made in this section of the river. However, adjusted data from the upper three areas shows that the relative order of mean concentrations in the three areas is not the same as that based on unadjusted data. After adjustment the areas of highest concentrations shifts from Lake Wallula to Lake Umatilla, and the difference between Lake Bonneville and Lake Wallula decreases.

Examination of adjusted data also allows an examination from a different perspective of the relationship between organochlorine concentration and sturgeon length. When DDE concentrations which have been adjusted to 7.09 percent lipids were plotted against sturgeon length (Figure 9) the correlation coefficient r becomes —.31 which is not significantly.(.05) different from zero. Also, adjusted PCB values plotted against length (Figure 10) yield a correlation coefficient r of — .02 which is not significantly.(.05) different from zero. Thus it appears that the correlation between length and concentration observed in the unadjusted data is due to the positive correlation between length and lipids (Figure 12).

adjustment to a common lipid value of 7.09 percent, the average in all sturgeon collected. sturgion collected from three sections of the Columbia River to mean concentrations after Comparison of mean concentrations of DDE and PGB's as they actually occurred in

A 700		Mean livid value	DDE	DUE DOM	CO	
O the same with	2	before adjustment	Before	After	Before	After
			ad justment	adjustment adjustment	Adjustment Adjustment	Adjustment
Lake Conneville	7	4.47	0,51	0,90	0.36	0.61
Texe Method	j,	\$ 9	1.42	2.30	1,02	1.57
	Vi	15.57	1.77	****	1.63	- 0

DISCUSSION

The monitoring information in this report is preliminary and quite limited. The sample size per river section does not allow the indepth data analysis that would provide conclusion one way or the other.

A number of items became apparent as the study proceeded and should influence future monitoring if it is undertaken. These items are, for most part, sample preparation, analytical analysis and the method in which the results are reported.

Our sampling technique was oriented toward edible filets to meet one of the study objectives. Since there is a positive correlation between lipid content and the quantity of organochlorine and PCB compounds in the sample it is possible to bias tissue samples by the amount of fat either left on or trimmed from the filet. There is also reason to believe that the analytical results regarding the residue levels found in the tissues and eggs which we submitted may be conservative. This was brought to light by seven sturgeon submitted by the Vancouver Fisheries Assistance Office to a laboratory other than the one which analyzed the tissues and eggs for this study. The seven sturgeon were from Lake Bonneville and were taken in the same general area where our samples were taken. The analytical results on these sturgeon indicated residue levels considerably higher than samples which we had submitted.

A cross check of the homogenates of those seven sturgeon by yet

another laboratory, U.S. Fish and Wildlife Service National Fisheries Research Laboratory, Columbia, Missouri (NFRL), also indicated residue levels higher than our analytical service but not as high as the laboratory which they were cross checking.

The method of reporting results also varied from laboratory to laboratory. Our analytical service reported PCB residues as Aroclor 1254 whereas NFRL reported PCB's as 1248, 1254, 1260, and total PCB's. The individual formulations could be important in determining the biodegradability and toxicity of the various compounds since more chlorinated PCB compounds generally are not as acutely toxic as the less chlorinated ones. In any event future work should include standardized reporting and quality control maintained over those laboratories providing analytical services.

Generally all three laboratories reported mean concentrations of PCB and DDT compounds below the 5 ug/g cut off level. Regard-less of the mean there were individual sturgeon which approached or exceeded, as reported by one laboratory, the cut off level.

As a precautionary measure it might be advisable to inform laymen of the tendency of PCB's to concentrate in fatty tissues, particularly immediately under the skin, and recommend that all fat be trimmed from sturgeon prior to cooking so as to reduce injestion of these compounds to a minimum.

it to unfortunate that we were unable to obtain more roe for analysis since this area appears to be one in which further investigation

is warranted. Collection of roe is difficult because few sturgeon under the six foot length contain eggs.

The Gonference Proceedings of the National Conference on Polychlorinated Biphenyls was used as a reference in obtaining comparative
PCB-fish relationships (Buckley, 1975). Jensen et al. (1970, cited
by Nebeker 1976) reported a possible relationship between PCB
residues in salmon eggs and egg mortality in Sweden. When residues
in groups of eggs ranged from 0.4 to 1.9 ug/g on a whole weight
basis (7.7 to 34 ug/g on a fat basis), related mortalities ranged
from 16 to 100 percent. This indicates that the threshold for egg
mortality was about 0.5 ug/g PCB. Such residues would be comparable to whole fish residues of 2.5 to 5.0 ug/g. Some of the residue
values reported in tissues which we submitted, and now thought to
be conservative, were above 2 ug/g and these samples consisted of
filets only. One egg sample collected in Lake Bonneville had a
reported PCB level of 1.45 ug/g (wet weight). This level is well
above the indicated 0.5 threshold mortality level for salmon.

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